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54 **Method of producing a thermoplastic tube having a smooth interior surface.**

57 A thermoplastic tube having a very smooth interior surface is produced by a method comprising: (1) extruding the thermoplastic tube whilst maintaining the interior surface in a molten state by a heating unit disposed within the area of a first mandrel near the nozzle of an extrusion mold, and (2) sliding the interior surface of the tube onto a second mandrel connected to the front end of the first mandrel through a heat-insulating material and in axial alignment with the first mandrel in a manner such as to bring the interior surface of the extruded tube into contact with the smooth surface of the second mandrel and simultaneously cooling the interior surface of the tube with a cooling medium.

1            METHOD OF PRODUCING A THERMOPLASTIC  
             TUBE HAVING A SMOOTH INTERIOR SURFACE

             This invention relates to a method of producing  
a thermoplastic tube, the interior surface of which is  
5 very smooth, so that it is useful as a piping material  
for handling ultra-pure water.

             A large amount of pure or ultra-pure water is  
used in the production process of semiconductor device  
(LSI) chips in semiconductor device manufacturing  
10 industries. Patterns of the size of approximately 2  $\mu\text{m}$   
are formed on the LSI chips, and thus impurities of  
0.2  $\mu\text{m}$  or more, which may be contained in the  
ultra-pure water used for the developing process of  
photoresists on the LSI chips, the etching process of  
15 LSI chips and the elimination process of the photo-  
resists from the LSI chips, cause breaking of LSI  
circuits thereby reducing the quality of the LSI chips  
and decreasing the yield. Particularly, organic  
matters, phosphorus compounds, etc., contained in the  
20 cells of bacteria, cause deterioration of the LSI chips.  
If pipes for transporting the ultra-pure water have  
irregularities on the interior surface, the water  
stagnates in the area of the irregularities, resulting  
in the growth of the bacteria.

25            Such pipes are usually made of rigid  
poly-vinylchloride, on the interior surface of which  
numberless irregularities having a height of  
approximately 10  $\mu\text{m}$  exist and bacteria increase  
30 therein, thereby requiring periodical cleaning thereof.

1 U.S. Patent 3,644,696 discloses an apparatus  
for smoothing the interior surface of a plastic hose,  
wherein a heating unit is positioned inside of the  
plastic hose obtained by extrusion molding and an  
5 induction coil which is concentric with the heating  
unit is positioned around the outside of the hose.  
The interior surface of the hose is heated by induction  
heating based on the heating unit and the induction  
coil to permit the surface tension of the plastics to  
10 smooth out the irregularities.

However, such a heating treatment of the  
interior surface of the hose merely allows acute angle  
portions in the irregularities to be rounded, and it  
15 cannot eliminate the irregularities themselves.  
Deformation of the irregularities due to the heating  
treatment is restored in the succeeding cooling step.  
Therefore, the smoothness desired for pipes  
transporting ultra-pure water therethrough cannot be  
20 obtained from such a heating treatment alone.

If the interior surface of the hose is heated  
to a high temperature which is sufficient to eliminate  
the irregularities, the plastics (i.e.,  
25 poly-vinylchloride) decomposes resulting in a drift  
therein so that hoses having a predetermined dimension  
cannot be produced continuously. Moreover, since the  
interior surface is at an exceedingly high temperature,  
it is insufficiently cooled in the succeeding cooling  
30 step and/or a temperature gradient in the direction of  
the thickness of the hose is great enough to produce  
residual strain, resulting in unstable dimensions of  
the hose.

1           In accordance with the present invention, there  
is provided a method of producing a thermoplastic tube  
which comprises (1) extruding the thermoplastic tube  
whilst maintaining the interior surface in a molten  
5 state by a heating unit disposed within the area of a  
first mandrel near the nozzle of an extrusion mold, and  
(2) sliding the interior surface of the tube on to a  
second mandrel connected to the front end of the first  
mandrel through a heat-insulating material and in axial  
10 alignment with the first mandrel in a manner such as to  
bring the interior surface of the extruded tube into  
contact with the smooth surface of the second mandrel  
and simultaneously cooling the interior surface of the  
tube with a cooling medium.

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The roughness of the surface of the additional  
mandrel is, in a preferred embodiment, 1.5-S or less.

Grooves are, in a preferred embodiment, formed in  
20 the circumferential direction on the surface of the  
second mandrel and a cooling medium is supplied under  
negative pressure, thereby ensuring that the tube comes  
into contact with the second mandrel. The  
circumferential grooves are, in a preferred embodiment,  
25 connected to each other by zigzag grooves formed  
therebetween, resulting in a path for the cooling  
medium, the said path being connected to a cooling  
medium feeder and/or a suction device, respectively,  
through holes in the bottom of each of the circumferential  
30 grooves.

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1           After passing over the second mandrel, the  
interior surface of the tube is, in a preferred embodi-  
ment, sprayed with a cooling medium.

5           The cooling medium is, in a preferred embodi-  
ment, water or air.

          Thus, the invention described herein makes  
possible the objects of (1) providing a method for the  
10 continuous production of a plastic tube which has a  
highly smooth interior surface thereby allowing the  
prevention of growth of bacteria thereon; (2) providing  
a method for the continuous production of a plastic  
tube which is useful as a piping material for  
15 ultra-pure water; and (3) providing a method for the  
production of a plastic tube which will remain clean  
for a long period with minimum washing.

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1           A thermoplastic tube of this invention is  
made of resin material such as poly-vinylchloride,  
polyethylene, polypropylene, etc., using an extrusion  
5           molding apparatus comprising an extruder and an extru-  
sion mold. The extrusion mold comprises an outer mold  
and a mandrel supported by a spider element within the  
outer mold in axial alignment with the outer mold to  
form a tubular cavity therebetween.

10           While a molten resin material is extruded  
from the nozzle of the extrusion mold through the  
tubular cavity into a tubular form, the interior  
surface of the extruded thermoplastic tube is melted by  
a heating unit disposed within the area of the mandrel  
15           near the nozzle of the extrusion mold.

          Generally, resin material to be extruded into  
a tubular form must be maintained at a certain  
temperature level or lower, at which the resin material  
20           is completely molten, since it (especially, poly-vinyl-  
chloride) tends to undergo thermal decomposition at  
temperatures above the melting point. Thus, a portion  
of the resin material is not molten, causing  
irregularities on the interior surface of the final  
25           product, i.e., the thermoplastic tube. According to  
this invention, the interior surface of the tube is  
maintained in a molten state under a temperature  
control by the heating unit while it comes out of the  
nozzle of the mold. The heating unit is located within

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1 the mandrel in the vicinity of the nozzle where the  
molten resin material forms a laminar flow, so that a  
limited area of the interior surface of the resulting  
tube or its surroundings is heated without undergoing  
5 thermal decomposition and thus the heated area is  
immediately cooled in the succeeding cooling step. As  
the heating unit, any heating unit known to be useful  
for melting the resin of this invention can be used, an  
example of which is an inexpensive electric heater by  
10 which a temperature control can be easily carried out.  
The temperature control can be carried out by detecting  
the temperature of the surface of the mandrel by a  
temperature sensor to regulate a voltage of a variable  
transformer depending upon the detected temperature of  
15 the surface of the mandrel. Wires for supplying  
electric power to the heating unit and for drawing  
signals from the temperature sensor are connected to a  
power source and a temperature detector, respectively,  
through the spider element supporting the mandrel in  
20 the extrusion mold.

The exterior surface of the mandrel on which  
the molten resin material passes is preferably of a  
"mirror-finished" hard chromium plating, a fluorocarbon  
25 polymer coating, etc.

The interior surface of the tube is then  
heated within the range of 180°C to 220°C when the tube  
is made of rigid poly-vinylchloride. If the tempera-  
30 ture is lower than 180°C, the interior surface of the  
tube is insufficiently molten so that a highly smooth  
surface cannot be obtained, even in any cooling  
condition, while the tube slides on the highly smooth  
exterior surface if an additional mandrel is applied.  
35 If the temperature is over 220°C, heat loss increases  
since the tube must be cooled in the succeeding cooling

1 step and, moreover, the tube tends to undergo thermal  
decomposition. The terminology "a highly smooth  
surface of the thermoplastic tube" means hereinafter  
the degree of the roughness of the surface of the tube  
5 which is 0.8-S or less (i.e., the roughness indicated  
by the quadruple triangle mark  $\overline{W}$ ) according to JIS  
B0601. The degree of this roughness can prevent  
stagnation and the growth of bacteria in irregularities  
on the interior surface of the tube.

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The interior surface of the tube which was  
heated on the mandrel by the heating unit is cooled  
while it slides on to the second mandrel. The  
roughness of the exterior surface of the second  
15 mandrel is preferably the same as that to be required  
for the interior surface of the tube, but a roughness  
of 1.5-S is sufficient because the roughness of the  
interior surface of the tube can be reduced to one-half  
of the roughness of the exterior surface of the  
20 second mandrel while the tube slides on the  
second mandrel. The exterior surface of the  
second mandrel is preferably of "mirror-finished"  
hard chromium plating or fluorocarbon polymer coating.

25 In order to bring the thermoplastic tube,  
which was extruded from the nozzle, into tight contact  
with the exterior surface of the second mandrel,  
negative pressure is applied through the  
circumferential grooves disposed on the exterior  
30 surface of the second mandrel. An alternative  
approach is to use the second mandrel of which  
external diameter is greater than that of the first mandrel,  
to enlarge the inside diameter of the thermoplastic  
tube extruded from the nozzle of the extrusion mold, to  
35 utilize a shrinking force in the tube. As the tube  
slides on the second mandrel in the condition where

1 it is in tight contact with the second mandrel, the  
interior surface of the tube is smoothed and  
immediately cooled to fix the smoothed surface. If the  
tube is allowed to stand at an insufficient cooling  
5 temperature it is liable to effect thermal deformation,  
and irregularities on the interior surface of the tube  
will be restored to such an extent that the interior  
surface of the tube will not contain the desired  
smoothness. Thus, the interior surface of the tube  
10 must be cooled to a low enough temperature while it  
slides on the second mandrel.

In order to cool the interior surface of the  
tube while sliding on the second mandrel, it is  
15 preferable to adopt a direct cooling technique  
attaining an excellent cooling effect in which a  
cooling medium such as water, air, nitrogen gas, etc.,  
is supplied between the interior surface of the tube  
and the exterior surface of the second mandrel to  
20 achieve excellent cooling of the interior surface of  
the tube. An indirect cooling technique in which the  
tube slides on the second mandrel which has been  
cooled by a cooling means can also be used. In order  
to bring the cooling medium into uniform contact with  
25 the tube in the circumferential direction according to  
the direct cooling technique, a plurality of circumferential  
grooves connected to each other by zigzag grooves  
therebetween are formed on the exterior surface of the  
additional mandrel to be filled with the cooling  
30 medium, which is recovered upon cooling the interior  
surface of the tube. The recovery of the cooling  
medium from the grooves can be attained by applying a  
positive pressure for supplying the cooling medium to  
the grooves or a negative pressure for sucking it from  
35 the grooves. The application of a negative pressure to  
the recovery of the cooling medium from the grooves is

1 advantageous over that of the positive pressure in that  
the negative pressure serves to draw the tube to the  
second mandrel. The amount of cooling medium  
required becomes greater as the degree of the negative  
5 pressure is increased. If the degree of the negative  
pressure is exceedingly high, frictional force between  
the tube and the second mandrel increase resulting  
in difficulty to take off the tube. Thus, the negative  
pressure is selected to be in the range of -500 mmHg to  
10 -600 mmHg. In such a manner, the cooling medium  
(especially, cooling water) must be recovered from the  
grooves, because if the cooling water remains in the  
tube, the interior surface of the tube is cooled and  
fixed ununiformly in the circumferential direction, re-  
15 sulting in a final product having less accuracy of size  
and containing flow marks from the cooling water on the  
interior surface.

The second mandrel for cooling the tube  
20 is connected to the front end of the first  
mandrel through a heat-insulating substance. ———

————— The pipe means, which  
is used to transport the cooling medium from a medium  
source to the grooves on the surface of the second  
25 mandrel through the spider element, is a hose made of a  
heat insulating substance such as a fluorocarbon resin  
which can be laid so that it can come into contact with  
the extrusion mold to be heated.

30 The cooling process during the slide of the  
tube on the second mandrel should be carried out at  
a sufficiently low temperature. Since the amount of  
cooling medium supplied into the second mandrel is  
limited to a certain quantity, production speed of the  
35 tube is limited to a certain level so as to cool the  
interior surface of the tube sufficiently. According

1 to this invention, nevertheless, the tube can be  
promptly produced since the interior surface of the  
tube is sprayed with a cooling water upon passing  
through the second mandrel, resulting in a highly  
5 smoothed surface thereof. According to experimental  
data, the interior surface of \_\_\_\_\_  
the tube must be cooled to a temperature of 110°C or  
less to attain a highly smooth level thereon, although  
the exact temperature depends upon the amount of  
10 sprayed cooling water.

The amount of sprayed cooling water must be  
small enough so that waterdrops are not produced since  
the drops on the lower portion of the interior surface  
15 of the tube cause not only cooling of the interior  
surface of the tube but also marks thereon. The amount  
of cooling water depends upon the inside circumference  
of the tube, and was approximately maximum 60 g/minute  
for the poly-vinylchloride tube having a nominal bore  
20 of 75 mm. and a thickness of 5.9 mm.

The cooling water can be sprayed from a jet  
nozzle under pressure or with compressed air. The  
latter is advantageous over the former in that  
25 sufficient cooling can be attained due to the  
additional cooling effect of the air jet.

Thus the extruded tube is cooled from inside,  
and then is cooled from outside when it passes through  
30 cooling water baths and is cut into a given length.

For a better understanding of the invention and  
to show how the same may be carried into effect, reference  
will now be made, by way of example, to the accompanying  
drawings in which:

35 Figure 1 is a schematic illustration of a method  
for the production of a plastic tube according to this

1 invention;

Figure 2 is an enlarged partly sectional side view showing an apparatus for the method of this invention; and

5 Figure 3 is an enlarged partly sectional side view showing another apparatus for the method of this invention.

Referring now to the accompanying drawings, Figures 1 and 2 show an extrusion mold 1 for a method of this invention, which comprises an outer mold 2 and a mandrel 3 supported by a spider element 4 within the outer mold 2 in axial alignment with the outer mold 2 to form a tubular cavity therebetween as an annular path 5 for molten resin material. The

15 extrusion mold 1 is connected to the front end of an extruder (not shown) in which a resin material is melted and mixed. The molten resin material is extruded from the nozzle 6 through the annular path 5 in the extrusion mold 1 into a thermoplastic tube form.

20 The resulting tube 7 is then cooled and formed into the desired shape by a forming mold (not shown) and a cooling bath (not shown) in a common manner.

The interior surface of the extruded tube 7

25 is kept molten by a heating unit 8 consisting of an electric heater which is disposed within the area of the mandrel 3 near the nozzle 6 and which is operated by means of an electric wire 9 through the spider element 4. A temperature-detecting sensor 10 is

30 located around the exterior surface of the mandrel 3 near the nozzle 6 to determine the temperature of the interior surface of the tube 7, thereby controlling a heating voltage to be applied to the heating unit 8 so that the temperature of the mandrel 3 can be

35 automatically regulated to a proper level. Numeral reference 11 is a wire for drawing signals from the sensor 10.

1           A second mandrel 12 is connected to the  
front end of the mandrel 3 by a screw means through a  
heat-insulating substance 13. On the surface of the  
second mandrel 12, four circumferential grooves 14,  
5 15, 16 and 17 are formed and zigzag grooves 18 are  
formed between the central grooves 15 and 16.  
Holes 19, 20 and 21 are formed in the bottom of each of  
the grooves 14, 16 and 17, respectively, and connected  
to a vacuum pump (not shown) outside the mold 1 through  
10 the inside of the mandrel 3 by means of a fluorocarbon  
polymer hose 22 to thereby maintain inside the  
additional mandrel a negative pressure. A hole 23  
(Fig. 2) in the bottom of the groove 15 is connected to  
a cooling water source outside the mold 1 by a  
15 fluorocarbon polymer hose 24 through which cooling water  
can be supplied thereto under pressure.

Compressed air and water are supplied by a  
hose 26 to a jet nozzle 25, which is positioned in  
20 front of the front end of the second mandrel 12,  
and water is jetted therefrom to the interior surface  
of the tube 7.

According to the above-mentioned mold 1, the  
25 tube 7 is extruded from the nozzle 6 while the interior  
surface thereof is molten on the exterior surface of  
the mandrel 3 heated by the heating unit 8, and then  
slides on the second mandrel 12 coming into tight  
contact therewith due to the drawing under negative  
30 pressure in the grooves 14, 16 and 17. While the  
cooling water flows from the groove 15 to the groove 16  
through the grooves 18, it contacts and cools the  
interior surface of the tube 7, and is removed from the  
hole 20 under negative pressure. By sliding the tube 7  
35 on the second mandrel 12 in such a manner that both  
are in tight contact with each other, the interior  
surface of the tube 7 is made highly smooth, and is  
immediately cooled. Upon passing through the

1 second mandrel 12, the interior surface of the  
tube 7 is sprayed with cooling water from the jet  
nozzle 25 in order to be cooled.

5 Figure 3 shows another extrusion mold 1 as  
another effective method of this invention, which  
comprises a cooling water-circulating path 27 near the  
surface of the second mandrel 12' instead of the  
grooves in Figures 1 and 2. To both ends of the  
10 path 27, a cooling water-inlet hose 28 and a outlet  
hose 29 are connected, respectively, to cool the  
second mandrel 12' with cooling water, thereby  
indirectly cooling the interior surface of the tube 7  
while the tube slides on the cooled second mandrel 12'.

15 The following Examples illustrate the invention.

#### Example 1

A molding apparatus, which consists of the  
extrusion mold shown in Figures 1 and 2 connected to  
20 the front end of a twin screw extruder having an  
opening diameter of 90 mm, was used for the formation  
of a rigid poly-vinylchloride tube, using a resin  
material of the following composition:

25 A rigid poly-vinylchloride resin for  
extrusion molding having a degree of polymerization  
1050 ... 100 parts by weight,  
organic tin-stabilizer ... 1.5 parts by weight,  
30 calcium stearate ... 1 part by weight,  
stearic acid ... 1 part by weight, and  
35 polyethylene wax ... 0.5 parts by weight.

Extrusion molding was carried out at an  
extrusion temperature (in the area between the extruder

1 and the mold) of 184°C at an extrusion rate of  
0.9 m/min to form a tube having a nominal bore of 75 mm  
(i.e., an outer diameter of 89 mm and a thickness of  
5.9 mm).

5 Extrusion tests were carried out at 180°C,  
190°C, 200°C and 210°C under a controlled voltage to  
the heating unit 8, in which the quantity of cooling  
water (at 16°C) to be supplied to the groove 15 in the  
10 \_\_\_\_\_ mandrel 12 was 200 g/min, 400 g/min and  
600 g/min and the quantity of sprayed water (at 16°C  
under a compressed air of 5 kg/cm<sup>2</sup>) from the jet  
nozzle 25 was 20 g/min, 40 g/min and 60 g/min.

The roughness of the interior surface of each  
15 of the resulting thermoplastic tubes was evaluated.  
The results are shown in Table 1 together with the  
temperature of the interior surface of each of the  
tubes just after passage through the additional  
mandrel.

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Table I

Temperature on the interior surface of the tube just after extrusion	Cooling water											
	200r/min			400r/min			600r/min			600r/min		
	20g/min	40g/min	60g/min	20g/min	40g/min	60g/min	20g/min	40g/min	60g/min	20g/min	40g/min	60g/min
180 °C	*	103 °C		*	78 °C		*	56 °C		*	56 °C	
	X	X	X	X	X	X	X	X	X	X	X	X
190 °C	*	118 °C		*	92 °C		*	68 °C		*	68 °C	
	X	X	X	X	X	X	X	X	X	X	X	X
200 °C	*	121 °C		*	98 °C		*	85 °C		*	85 °C	
	X	X	X	X	X	X	X	X	X	X	X	X
210 °C	*	130 °C		*	106 °C		*	94 °C		*	94 °C	
	X	X	X	X	X	X	X	X	X	X	X	X

Note: The mark O indicates the roughness of the surface at 0.8-S or less and the mark X indicates the roughness of the surface at greater than 0.8-S.

\* : Temperature on the interior surface of the tube just after passing through the second mandrel.

1 Example 2

Using the same molding apparatus as in Example 1 and cooling air instead of cooling water, extrusion molding was carried out in the same manner as  
5 in Example 1, resulting in a thermoplastic tube having the desired highly smoothed interior surface.

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1 CLAIMS:

1. A method of producing a thermoplastic tube,  
which comprises:

5 (1) extruding the thermoplastic tube whilst main-  
taining the interior surface in a molten state by a  
heating unit disposed within the area of a first  
mandrel near the nozzle of an extrusion mold, and  
10 (2) sliding the interior surface of the tube on to a  
second mandrel connected to the front end of the  
first mandrel through a heat-insulating material and  
in axial alignment with the first mandrel in a  
manner such as to bring the interior surface of the  
extruded tube into contact with the smooth surface  
15 of the second mandrel and simultaneously cooling the  
interior surface of the tube with a cooling medium.

2. A method according to Claim 1, wherein the  
roughness of the surface of the second mandrel is 1.5-S  
20 or less.

3. A method according to Claim 1 or 2, wherein grooves  
are formed in the circumferential direction on the surface  
of the second mandrel and a cooling medium is supplied  
25 under a negative pressure, thereby ensuring that the tube  
comes into contact with the second mandrel.

4. A method according to Claim 3, wherein said  
circumferential grooves are connected to each other by  
30 zigzag grooves formed therebetween, resulting in a path  
for the cooling medium, said path being connected to a  
cooling medium feeder and/or a suction device, respectively,  
through holes in the bottom of each of the circumferential  
grooves.

35 5. A method according to Claim 1, 2, 3 or 4, wherein  
after passing over the second mandrel, the interior surface

1 of the tube is sprayed with a cooling medium.

6. A method according to any preceding claim,  
wherein said cooling medium is water or air.

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FIG. 1

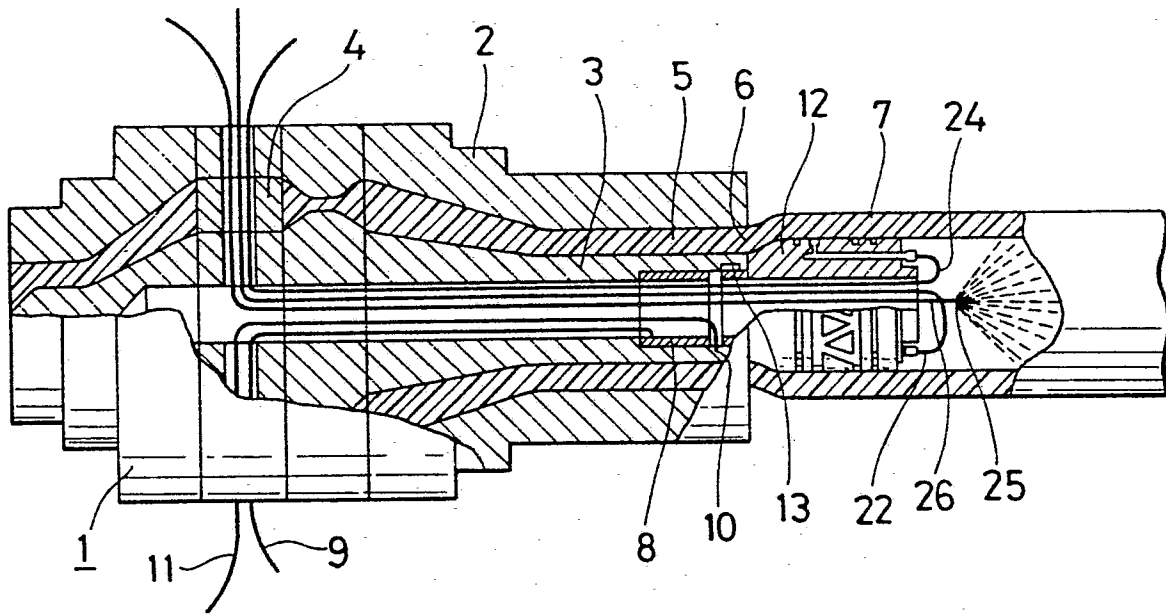


FIG. 2

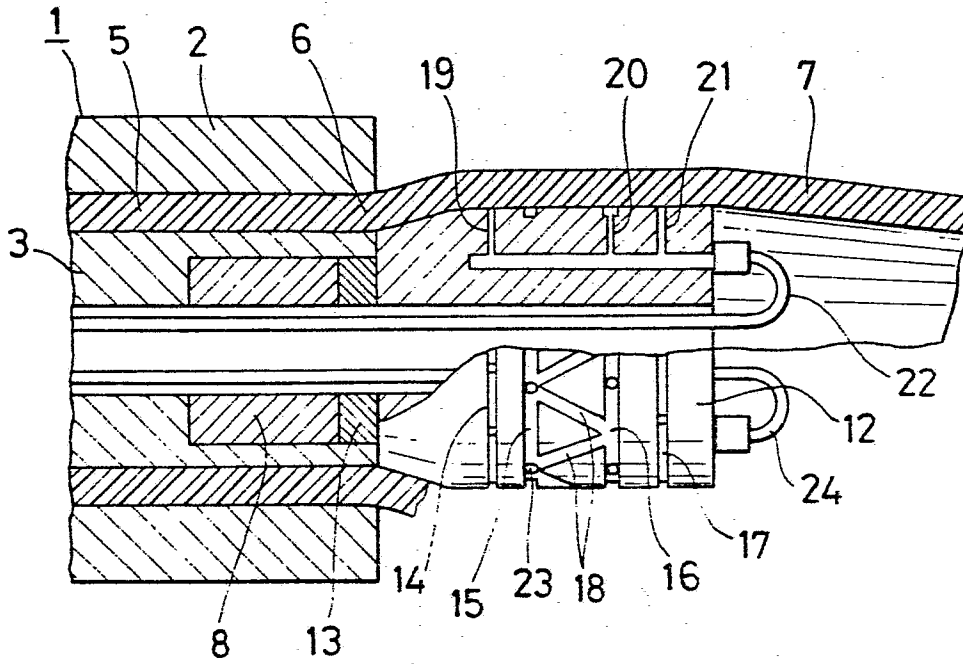


FIG. 3

